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BIO-OIL FROM FAST PYROLYSIS OF OIL PALM EMPTY FRUIT BUNCHES

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Abstract: *This study is an investigation on fast pyrolysis technology of oil palm empty fruit bunches (EFB) to bio-oil. EFB is one of the solid wastes that are rapidly increasing in the palm oil industry. The composition and particle size distribution of the unwashed feedstock and washed feedstock were determined and its thermal degradation behaviour was analysed by thermogravimetric analysis (TGA). A 150 g/h fluidized bed bench scale fast pyrolysis unit was used to study the impact of key variables: reactor temperature in the range of 425°C to 550°C and feedstock ash content in the range of 1.15 to 5.43 mf wt%. The properties of the liquid product were analysed and compared with wood derived bio-oil and petroleum fuels. It was found that the maximum ash content of washed feedstock that produced homogenous liquids is less than 3 mf wt%. The results of pyrolysis experiments showed that the bio-oil from washed EFB with low ash content had similar properties as wood.*

Keywords: empty fruit bunches, fast pyrolysis, oil palm, bio-oil, ash, washed feedstock, thermogravimetric analysis

1. INTRODUCTION

Fast pyrolysis is a thermal decomposition process that occurs at moderate temperatures in which the biomass is rapidly heated in the absence of oxygen or air to produce a mixture of condensable liquids, gases and char.[1] The process of fast pyrolysis is one of the most recent renewable energy processes which promises high yields of liquid with minimum of gas and char if this process is carefully controlled. Normally for woody feedstocks, temperatures around 500ºC together with short vapour residence times are used to obtain bio-oil yields of around 70%, and char and gas yields of around 15% each.[2] Bio-oil can be used as a substitute for liquid fossil fuels in some applications because this liquid is a high density oxygenated liquid. It can be burned in diesel engines or boilers, though further work is still required to demonstrate long term reliability.[3] It can possibly be used for the production of speciality chemicals, currently mainly flavourings. Renewable resins and slow release fertilizers are other potential applications, which have been the subject of research.[4]

The fast pyrolysis process is perceived to offer logistical and hence economic advantages over other thermal conversion processes.[4] This is because the liquid product can be stored until required or readily transported to where it can be most effectively utilized, and the liquid's density is also high (around 1.2 kg/litre[2]). Although Malaysia is the world's largest producer and exporter of palm oil, it also generates large quantities of oil palm wastes. Therefore, an investigation on fast pyrolysis technology to convert oil palm waste such as EFB to bio-oil is important. The bio-oil derived from unwashed EFB were presented elsewhere[5] and it was found that in all cases the liquid product separated into two phases creating difficulties in fuel applications. In this work, washed EFB was pyrolysed in a fluidized bed reactor with a nominal capacity of 150 g/h, with the objective of determining the effect of ash reduction on yield and the maximum ash level giving a homogenous bio-oil. The results are compared to those obtained from pyrolysis of various feedstocks with different ash contents.

2. FEEDSTOCK PREPARATION AND PROPERTIES

The EFB used in the experiments was supplied by Malaysian Palm Oil Board (MPOB). Samples received were relatively dry having less than 10 mf wt% moisture, and were in the form of whole bunch. Particle size reduction was required to allow fast pyrolysis of the EFB on the available 150 g/h system. The pyrolysis experiments on unwashed and washed feedstocks were carried out. The results were divided into two parts: pyrolysis of unwashed feedstock and pyrolysis of washed feedstock. However, the results of pyrolysis on unwashed EFB were presented elsewhere.[5]

The process of pyrolysis is complex, but the most accepted theory is that primary vapours are first produced. These primary vapours then further degrade to secondary tars, char and gases, and this degradation can be enhanced by catalysis, high temperature and longer residence time. High ash in biomass generally promotes secondary reactions of primary pyrolysis products since some ash components, primarily potassium and sodium, are known to be catalytically active.[6,7] Therefore, secondary reactions should be avoided for the production of liquid.

The bunches were first manually chopped into smaller pieces so that they could be fed into a shredder. Subsequently, a Fritsch grinder with a screen size of 500 µm was used to reduce the EFB size to less than 500 µm. The distribution of feed particle size after grinding is given in Table 1. The ash content of each size fraction was determined using National Renewable Energy Laboratory (NREL) Standard Analytical Method LAP005, and the mass average of the size fractions of ash is 5.39% compares well, within the accuracy of the measurements, with the original sample sent by MPOB, which had an ash content of 5.36%.

After extensive feeding trials, it was found that only particles between 250 to 355 µm were easily fed. Both of the size fractions below and above this range frequently led to blockage of the feeder. Empty fruit bunches had a much higher bulk density than other biomass types. Therefore, feed particles tended to stick together, thus, making the feeding of the reactor difficult. Further details can be found in Abdullah.[8]

Table 1: Particle size distribution of EFB

Feed particle size (µm)	Mass fraction	Average ash (mf wt %)
less than 250	22	7.44
250–355	30	5.29
355–500	42	4.82
more than 500	6	4.72
mass average	-	5.39

The key properties of EFB, both measured for this research and from literature, are given in Table 2. The high ash and potassium values are noteworthy, as it is well known that ash, and in particular potassium, lead to reduced liquid yields in fast pyrolysis.[3] The carbon and hydrogen contents are comparable to woody biomass, as is the measured heating value. The lowest high heating value HHV in the literature[9] may be due to confusion between values quoted on a dry basis as opposed to a wet basis, a problem apparent elsewhere in the literature, for example Yusoff:[10] a value of 10 MJ/kg is quoted for dry oil palm matter, which is clearly too low for a ligno-cellulosic biomass on a dry basis, or Husin:[11] values for wet fresh fruit bunches (FFB) are used for dry FFB.

Table 2: Properties of EFB (mf wt%)

Component / Property	Literature values	References	Measured	Method
Cellulose	59.7, 38.1–42.0	12, 13	-	-
Hemicellulose	22.1, 16.8–18.9	12, 13	-	-
Lignin	18.1, 10.5–11.7	12, 13	-	-
Elemental analysis				
Carbon	48.9, 48.8, 49.2–50.6	14, 15, 16	49.07	Elemental analyser
Hydrogen	7.33, 6.3	14, 15	6.48	
Nitrogen	0.0, 0.7, 0.78–1.19, 0.2, 0.8, 0.44	14, 15, 16, 17, 9, 18	0.7	
Sulphur	0.68, 0.2	14, 15	<0.10	
Oxygen	40.2, 36.7	14, 15	38.29	By difference
K	2.41, 2.24	9, 18	2.00	Spectrometry
K_2O	3.08–3.65	16	-	-
Proximate analysis			-	-
Moisture	-	-	7.95	ASTM E871
Volatiles	87.3, 75.7	14, 15	83.86	ASTM E872
Ash	3.02, 7.3, 4.3	14, 15, 9	5.36	NREL LAP005
Fixed carbon	9.6, 17	14, 15	10.78	By difference
HHV (MJ/kg)	19.0, 17.86, 15.5, <10	14, 19, 10, 9	19.35	Bomb calorimeter
LHV (MJ/kg)	17.2	18	-	-

3. EXPERIMENTAL PROCEDURE

A fluidized bed bench scale fast pyrolysis unit operating at atmospheric pressure was employed for all runs. Figure 1 shows a schematic diagram of this unit, which consists of three main parts, namely the feeder, reactor and product collection system. The reactor is a stainless steel (type 316) cylinder of length

260 mm and diameter 40 mm. The heating medium in the reactor is inert sand of particle size 355 to 500 μm. The sand fills the reactor to a height of approximately 8 cm and expands during fluidization to 12 cm. The fluidizing gas is nitrogen, which is preheated prior to entering via the base of the reactor.

The char is elutriated out of the reactor by the fluidizing gas flow, which is known as "blow-through" mode.[20] The char is then separated from the product stream in a cyclone. The condensable vapours are collected in the liquid products collection system, which consists of two cooling condensers, an electrostatic precipitator and a cotton wool filter. The incondensable gases leave the system through a gas meter and are then sampled by gas chromatography (GC) to assess the quantity and type of gas produced.

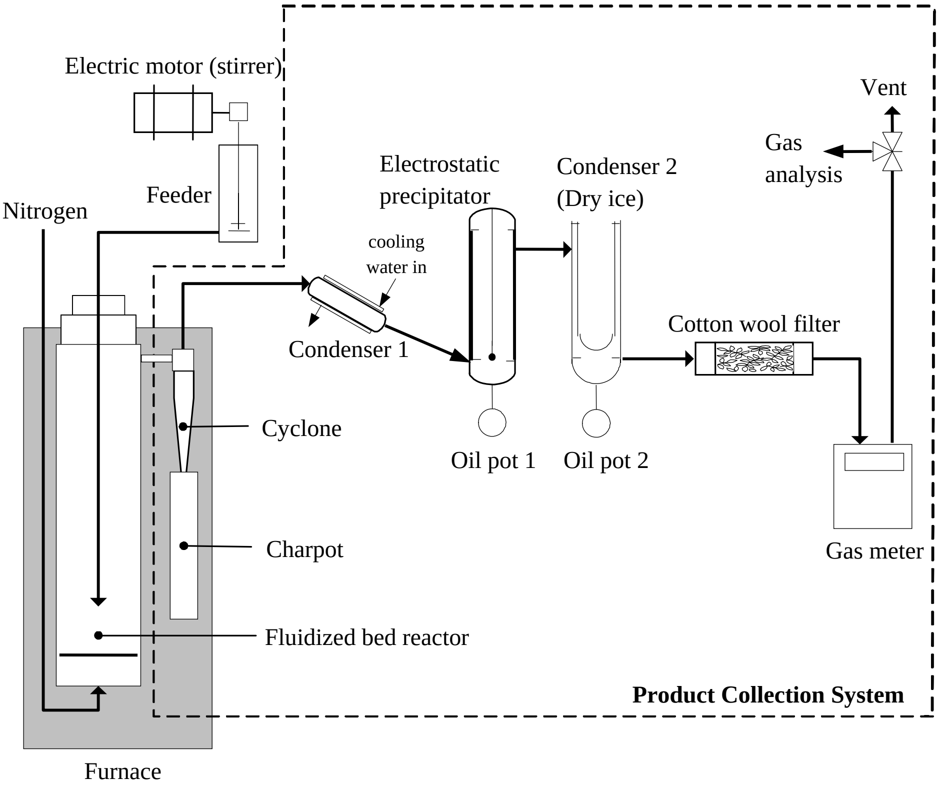


Figure 1: 150 g/h fluidized bed pyrolysis system

4. EXPERIMENTAL PLAN

A series of unwashed and washed feedstocks have been pyrolysed over a range of temperatures from 425°C to 550°C on EFB feedstock of size 355 to 500 µm. In all cases, the maximum yield of organics has been determined along with the temperature at which the maximum yield occurs. The experimental data from the runs are arranged in order of increasing temperature and the pyrolysis temperature chosen to cover this range is approximately 25°C. A number of washed feedstocks with different ash content have been pyrolysed in order to determine the maximum ash level that produce the homogenous pyrolysis liquids. The results are compared to those from pyrolysis of various feedstocks with different ash contents.

In order to investigate the effect of temperature, fast pyrolysis experiments were carried out at a vapour residence time of 1.01 to 1.04 s on feedstock of size 355 to 500 µm covering the temperature range of 425°C to 550°C in 25ºC increment. The ash content of feedstock used for all run was 1.03 mf wt%.

Table 3: Washing procedure to reduce the ash content of EFB

Ash content (mf wt%)	Water washing method
1.03	Soak 100 g of feedstock of size 250–355 µm for 24 hours at ambient temperature in 7 litre distilled water
2.14	Soak 100 g of feedstock of size 2–3 cm for 20 minutes at ambient temperature in 5 litre distilled water
3.05	Soak 100 g of feedstock of size 2–3 cm for 10 minutes at ambient temperature in 5 litre distilled water
3.68	By manual agitation of 100 g of feedstock of size 2–3 cm for 1 minute at ambient temperature in 5 litre distilled water
5.43	Unwashed feedstock that was not subjected to any washing

In order to study the effect of ash content on products yields, five experiments were carried out at reactor temperature of 500ºC and hot vapour residence times of 1.02 to 1.06 s with feedstock of 1.03, 2.14, 3.05, 3.68 and 5.29 mf wt%. The washed feedstocks used were subjected to washing process as

shown in Table 3. All samples were grounded to a specific size before the pre-treatment.

The pyrolysis experiment was carried out at a temperature of about 500°C and at the residence time of 1.1 s on the washed feedstocks of size 355 to 600 µm with ash content of 1.06 mf wt% using the 1 kg/h rig in order to study the different product collection system of the 1 kg/h rig.

5. THERMOGRAVIMETRIC ANALYSIS OF WASHED EFB AND UNWASHED EFB

The thermal characteristics of washed and unwashed EFB were analysed with a computerized Perkin-Elmer Pyris 1 thermogravimetric analyser. Thermogravimetric analysis (TGA) was performed under 100 ml/min nitrogen with a heating rate of 10ºC/min. The thermal degradation characteristics of unwashed and washed feedstocks are displayed in Figures 2 and 3 by thermogravimetry (TG) and differential thermogravimetry (DTG) curves, respectively. Figure 2 shows the TG curves or fraction of weight loss as a function of temperature for samples of various ash content. From 100 to 300°C, it shows mainly the thermal degradation of extractives. The weight loss for all feedstocks was insignificantly changed from temperatures of 100°C to 270°C. From 270°C to 400°C the weight loss is the highest for all feedstocks, and it may be due to the thermal degradation of polymer blocks of biomass (such as hemicellulose, cellulose and lignin). And above 400°C the loss can still be observed as the more difficult thermal degraded compounds are present.

Figure 2 also illustrates that the total weight loss between 100°C to 450°C is 77.4%, the highest for the feedstock of the lowest ash content. Figure 4 shows the weight loss of all feedstocks for the temperature range. It was also found that the decomposition rate at the temperature higher than 450°C is faster for the feedstock of the lowest ash content, but was slower for the unwashed feedstock which had the highest ash content.

Figure 3 shows DTG curves represented by derivative weight loss as a function of temperature. The figure shows the peak shifted to higher temperature as ash content in the feedstock was reduced by washing. It is well known that ash containing minerals exerted a catalytic action during thermal decomposition of polymer blocks,[21] easing hemicellulose/cellulose decomposition to lower temperatures. Yang et al.[22] had previously studied that decomposition of hemicellulose, cellulose and lignin were from 220 to 300°C, 300 to 340°C and 750 to 800°C, respectively. Possibly explained the various peaks appearing from 250 to 400°C representing hemicellulose, cellulose and lignin degradation for the

feedstocks. This result is in agreement with the previous studies done by Raveendran et al.[23,24] and Jensen et al.[25] They also showed that clear temperature shifts were observed for several herbaceous biomasses by reducing inorganic content through water washing.

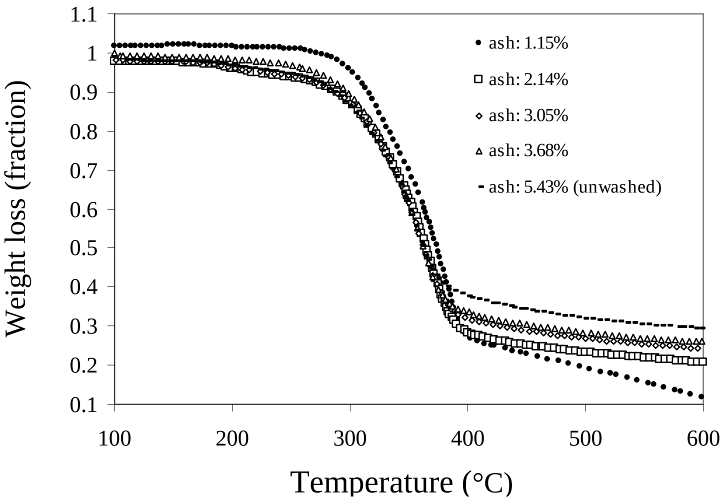


Figure 2: TG curves of unwashed and washed feedstock

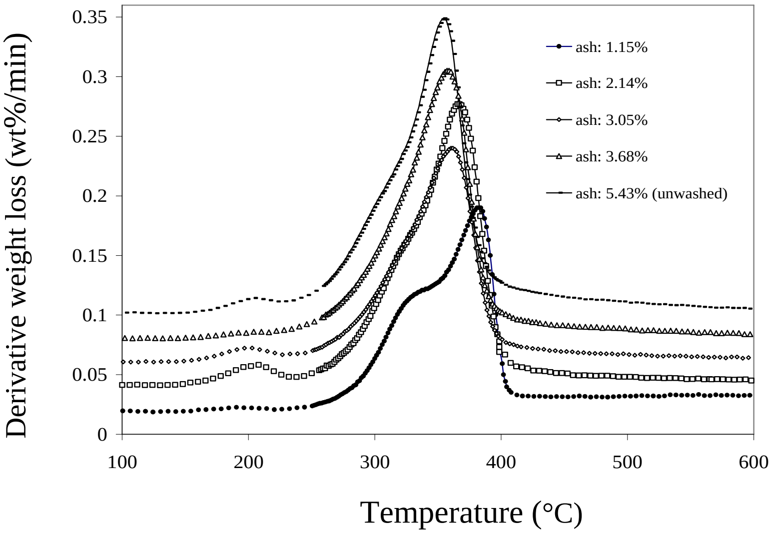


Figure 3: DTG curves of unwashed and washed feedstock

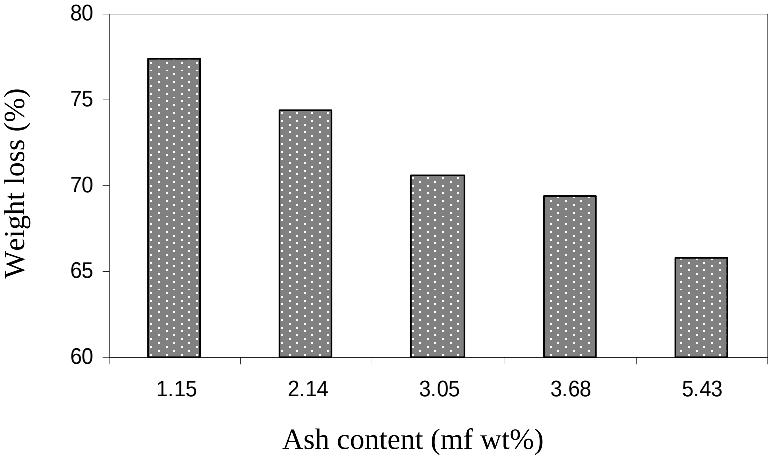


Figure 4: Weight loss in TGA between the temperatures of 100°C to 450°C for samples of various ash content

6. RESULTS AND DISCUSSION

6.1 Effect of Reactor Bed Temperature on Product Yields

The results of pyrolysis experiments are included in the graph of the major products plotted in Figure 5. All yields are given on a dry feedstock basis. The gas yield rises with temperature, while the char yield decreases. The water yield is approximately constant over the temperature range considered, while there is a maximum for the organics and liquids yields of 61.3% and 72.4%, respectively at about 500°C. The product yields as shown in Figure 5 are not significantly changed with the increase of pyrolysis temperature. It is possible that since EFB has already undergone sterilization and stripping processing in order to separate the sterilized oil palm fruit from the sterilized bunch stalks, therefore the cellular or molecular structure has been weakened and this has made it easier to pyrolyse as it is more susceptible to thermal degradation. The ash content of the feedstock used in this experiment is 1.03 mf wt%. The method used to remove the ash from the feedstock is described in Table 3. Figure 6 shows that the yield of carbon dioxide is relatively constant while carbon monoxide yield increases with temperature increase. Overall it appears that the washed feedstocks gave higher yields of organics at the expense of char and gas.

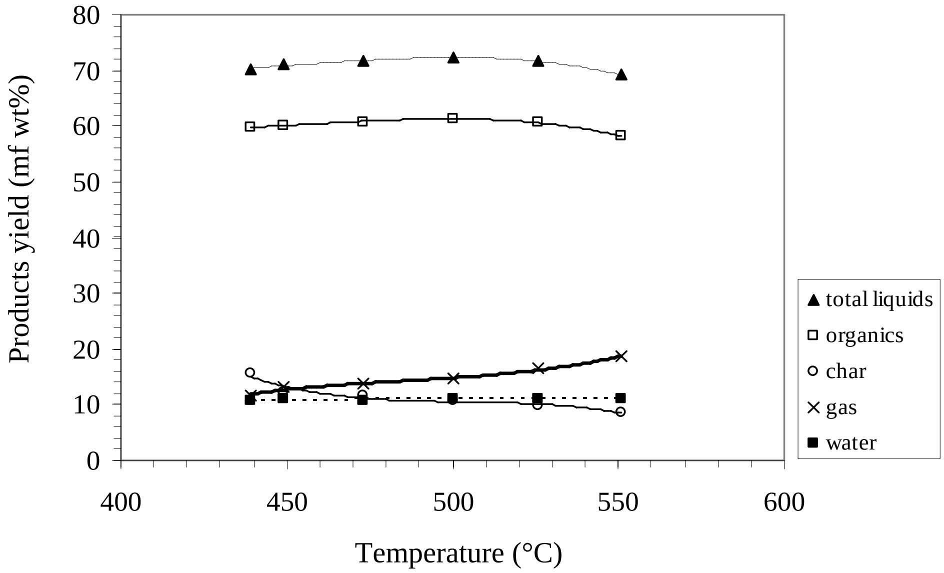


Figure 5: Products yield as function of temperature for washed feedstock of ash content 1.03 mf wt%

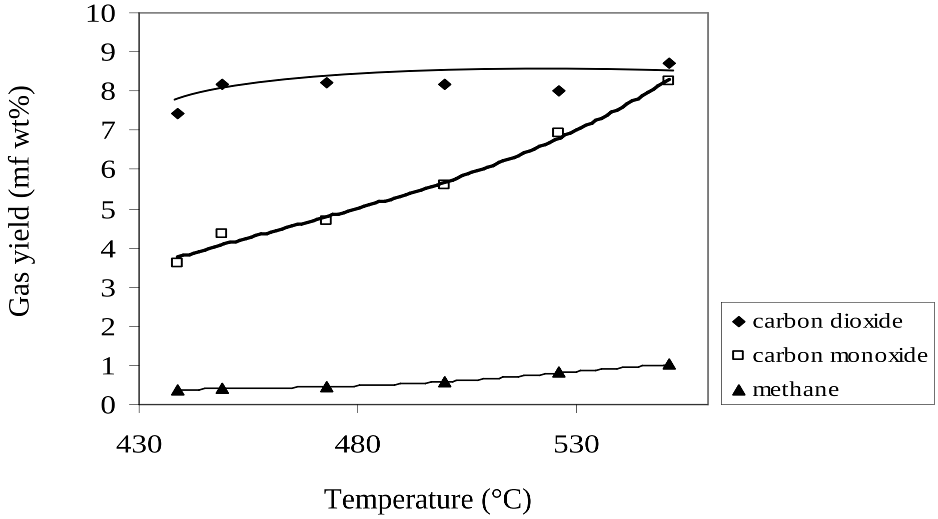


Figure 6: Gas yields as function of temperature for washed feedstock of ash content 1.03 mf wt%

6.2 Effect of Ash Content of Feedstock on Product Yields

Figure 7 shows that the organics yield achieved up to 61.3 mf wt%. This brings the total liquids up to 72.4 mf wt% for the washed feedstock of ash content 1.03 mf wt%. Overall it shows that the yield of organics decreased while the char yields and gas yields increased as the feedstock ash content increased. As mentioned earlier, it is generally accepted that ash, and particularly potassium, is catalytically active and favours secondary reactions leading to a reduction in organics yield, and corresponding increases in reaction water, gas and char yields.[3,26] The water yields were found to remain relatively constant throughout the ash content range investigated. It was found that the maximum ash content of washed feedstock that produce homogenous liquids (single phase oil) was less than about 3 mf wt%.

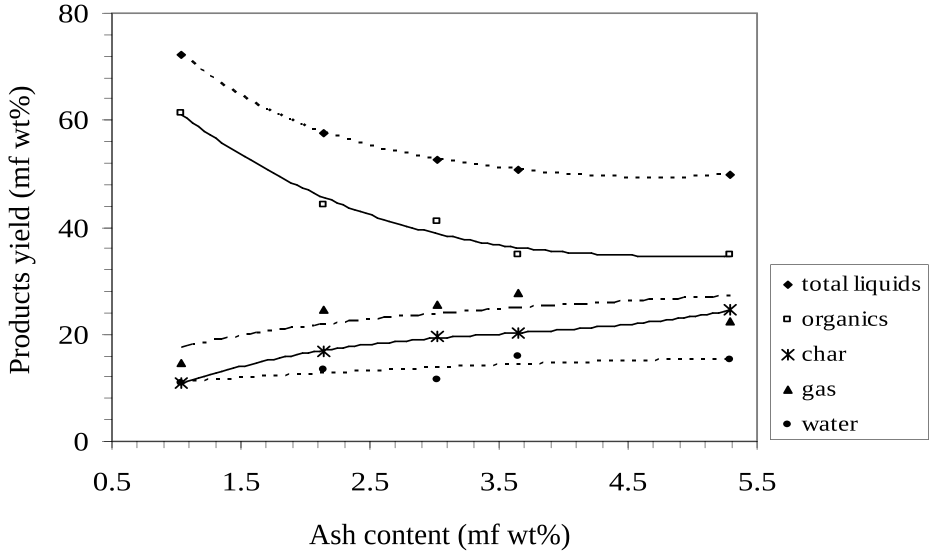


Figure 7: Effect of ash content on products yield

6.3 Feedstock Comparison

The results of pyrolysis experiment at the temperature of 400°C to 650°C on unwashed EFB, washed EFB and other type of feedstocks from published sources are shown in Table 4.

Poplar aspen[27] is a wood, which has low ash content of 0.39 mf wt% and high cellulose content of 42.3 mf wt%, while EFB, corn stover[6] and wheat straw[6] are agricultural residues which have ash content of 5.29, 11.0 and 4.6 mf wt%, respectively. It is evident that a woody poplar aspen, which has low ash content, produced the best organics yield than non-woody such as corn stover, and wheat straw which has a higher ash content. It can be seen that washed EFB, which has the ash content of 1.03 mf wt%, produced the organics yield of 61.34 mf wt% while organics yield of poplar aspen is 58.9 mf wt%.

Figure 8 illustrates the yield of organics from pyrolysis for a range of feedstocks from different sources as mentioned in Table 4. The organic yield percentage of washed EFB is almost the same as poplar aspen, while the organic yield percentage of unwashed EFB is almost the same as wheat straw and corn stover, which have higher ash content. This showed that washed EFB with low ash content has similar properties as wood-like poplar aspen. It is also evident that as the ash content of the feedstock increases, the yield of organics decreases.

Table 4: Comparison of pyrolysis products from a range of feed stocks at 500°C

Feedstock	Unwashed EFB	Washed EFB	Poplar aspen	Corn stover	Wheat straw
Reference	this work	this work	27	6	6
Cellulose (mf wt%)	59.7	not determined	42.3	31.0	32.4
Hemicellulose (mf wt%)	22.1	not determined	31.0	43.0	41.8
Lignin (mf wt%)	18.1	not determined	16.2	13.0	16.7
Moisture (mf wt%)	7.48–8.96	6.04–6.54	5.0	6.5	9.0
Ash (mf wt%)	5.29	1.03	0.39	11.0	4.6
Yields (mf wt%)					
Char	24.52	10.76	11	42.2	22
Gas	22.31	14.70	14	13.7	19
Organics	34.71	61.34	58.9	27.3	37
Total liquids	49.8	72.36	74.1	43.1	54

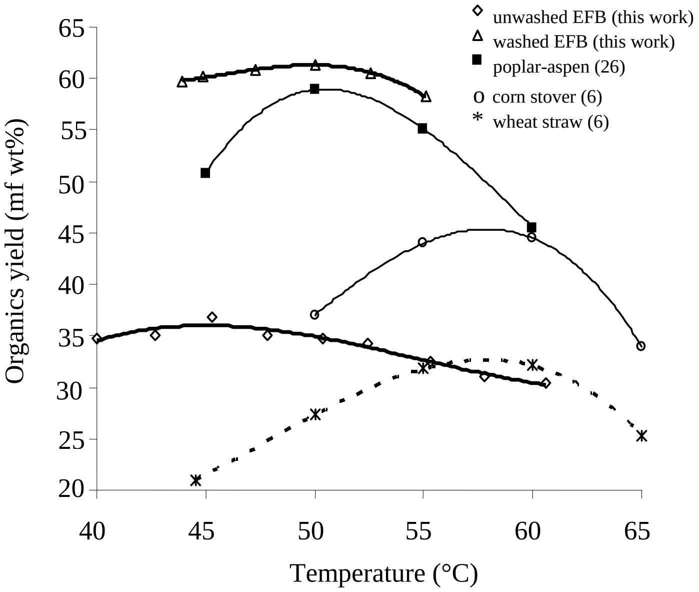


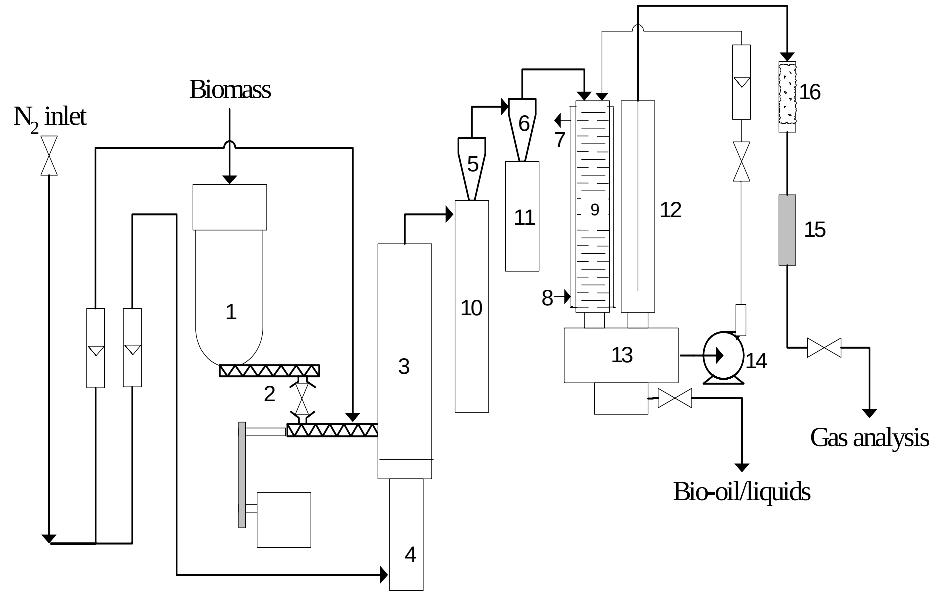
Figure 8: Yield of organics from pyrolysis of a range of feedstocks from different source

6.4 1.0 kg/h Reactor

Figure 9 shows the schematic diagram of the 1 kg/h fluidized bed reactor consisting of a stainless steel reactor, a fluid bed gas heater chamber, a feed container with screw feeder, a solid-gas cyclone separator, a quench column and liquid collectors. Working principle of a 1 kg/h rig is the same as the 150 g/h rig which employs nitrogen as the fluidizing gas and sand as the bed particles. The sand bed was heated to a desired temperature of about 500°C. The washed feestocks of size 355 to 600 µm were fed into the bubling hot sand in the reactor at a controlled feed rate. The reaction products (product vapours) were passed through two cyclones where the char was removed before being quenched with cooled recycle isopar. Once the vapours product passed through the quench column, the condensed liquid was collected by liquid collector (tank). The vapours were finally passed through an electrostatic precipitator (EP) which was very effective in capturing the vapours. The EP was present after the quench column. The pyrolysis liquid ran down the walls of the EP and accumulated at the bottom which was attached to the tank as well. The isopar in the tank was pumped into the quench column to cool and capture the vapours. The tank which was attached at the bottom of quench column and EP was the main liquid collection. It was found that a dark brown sticky mass continued to stick on the wall of EP although the pyrolysis experiment was carried out using 1 kg/h rig.

The amount of dark brown stuck mass stuck on the EP's wall and liquids collected in the tank was about 22.8% and 74% of total pyrolysis liquids produced from pyrolysis experiment, respectively.

Table 5 shows the results of liquids produced using the 150 g/h rig in comparison with the results of liquids produced using 1 kg/h rig. Both experiments were carried out with similar run condition but on different capacity of reactor. Generally, the results from both experiments were comparable. However, for liquids produced using the 1 kg/h rig, the organics yield was slightly lower, the char yield and gas yield were slightly higher than the liquid produced using the 150 g/h rig. It may be due to more secondary reactions occurred in the liquid produced using 1 kg/h rig, as the residence time was higher (1.1 s) than for liquids produced using the 150 g/h rig (1.02 s), thus, producing more char and gas yield.



Legend:

1. Feed container
2. Screw feeder
3. Fluidized bed reactor
4. Gas heater
5. Cyclone 1
6. Cyclone 2
7. Cooling water out
8. Cooling water in
9. Quench column
10. Char pot 1
11. Char pot 2
12. Electrostatic precipitator (EP)
13. Tank
14. Pump
15. Cotton filter
16. Dry ice condenser

Figure 9: 1 kg/h fluidized bed pyrolysis system

Table 5: Comparison of pyrolysis liquid produced from 150 g/h rig and 1 kg/h rig

Reactor	150 g/h	1 kg/h
Reactor temperature (°C)	500	499
Hot vapour residence time (s)	1.02	1.10
Feedrate in wet basis (g/h)	70.17	1247.11
Feed moisture (mf wt%)	6.04	7.25
Ash (mf wt%)	1.03	1.06
Particle size (μm)	355–500	355–600
Run time (min)	64.33	52.23
Input (g)		
Wet feed	75.23	1085.61
Dry feed	70.95	1012.25
Water in feed	4.28	73.36
Yields (mf wt%)		
Char	10.76	11.19
Organics	61.34	57.25
Gas	14.70	23.07
Water	11.01	6.96
Total liquids	72.36	64.21
Closure	97.8	98.47
Gas (mf wt%)		
Methane	0.58	0.63
Carbon dioxide	8.17	13.17
Carbon monoxide	5.59	8.77
Hydrogen	0.07	0.03
Ethylene	0.17	0.27
Ethane	0.10	0.12
Propane	0.02	0.04
Propylene	0.01	0.01

6.5 Physical and Chemical Properties of Products

The pyrolysis liquids produced homogenous liquids if the ash content of washed feedstock is less than about 3 mf wt%. A comparison of key properties for the two phases produced from unwashed feedstock, homogenous liquids from washed feedstock, with those of wood derived bio-oil, light fuel oil and heavy fuel oil are given in Table 6, which also contains the ultimate analysis of the liquid products.

Table 6: Characteristics of pyrolysis oil compared to petroleum fuel[3]

	Unwashed EFB		Washed EFB		Wood derived bio-oil	Light fuel oil	Heavy fuel oil
	Organics phase	Aqueous phase	Liquid from EP	Liquid from tank			
C	69.35	13.83	56.47	41.86	32–48	86.0	85.6
H	9.61	11.47	7.85	7.82	7–8.5	13.6	10.3
N	0.74	0.14	0.22	0.1	<0.4	0.2	0.6
O	20.02	74.56	35.46	50.22	44–60	0	0.6
S	-	-	-	-	<0.05	<0.18	2.5
Moisture content	7.9	64.01	6.66	21.68	20–30	0.025	0.1
HHV (MJ/kg)	36.06	-	25.29	18.63	-	-	-
LHV (MJ/kg)	-	-	-	-	13–18	40.3	40.7

7. CONCLUSIONS

A series of washed EFB have been successfully fast pyrolysed using a 150 g/h fluidized bed reactor operating at atmosphere pressure. The maximum yield for liquids produced from washed EFB was increased to be around 72% compared to liquids produced from unwashed EFB, which was about 55%.[5] The ash content of feedstock has been found to influence the yield of organics. The higher the concentration of ash in the feedstock the lower the yield of pyrolysis liquid. This showed that washed EFB with low ash content has similar properties as wood. Reduction of ash content to less than about 3 mf wt% is required in order to produce homogenous liquids or to avoid phase separation of liquids.

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